LAB MANUAL

FUEL TESTING AND CHEMICAL ANALYSIS

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List of Experiments

1. To determine the Proximate analysts of coal.
2. To determination of Cu % in brass by electrochemical analysis Method.
3. Determination of flash point and fire point of a given Liquid fuel.
4. To prepare the iron ore Sample and determine the iron content of that Sample by Potassium dichromate method.
5. To determine two percentage of Calcium and magnesium in dolomite ore.

Experiment-1

AIM OF THE EXPERIMENT:-

To determine the proximate analysis of coal**.**

APPARATUS REQUIRED:-

1. Coal
2. Digital balance
3. muffle furnace
4. Silica crucible**.**

THEORY:-

Determination of moisture, volatile matter, ash and fined. Carbon is coal comprises its proximate analysis.

Determination of moisture content in coal: Loss in weight of coal caused by heating of weighed quantity of coal simple for one hour at 105°c. Is the moisture content in coal.

%moisture is given bymoisture in coal $= \frac{\text{loss is weight of coal/Weight of}}{Initialweightofcoal} \times 100$

Determination of volatile matter in Coal: It is the loss in weight of moisture face Powdered coal when heated in a crucible fitted with cover in a muffle furnace at 950°C for 7 minutes.

%Volatile matter $= \frac{\mathbf{LossinWeightofmoisturefreecoal}}{\boldsymbol{weightofmoisturefreecoal}} \times \mathbf{100}$

Determination of ash in coal: It is the weight of residue obtainedafterburning a weighed quality of coal in an open crucible at 750°C in Mufflefurnacetill a constant weight is achieved.

Ash in Coal$= \frac{\mathbf{weightofresiduceashformed}}{\mathbf{weightofcoalinitallytaken}} \times \mathbf{100}$

Determination of fixed carbon: It is determined indirectly by deducting the sum total of moisture,volatile matter and Ash percentage from 100.

%fixed carbon in coal= 100-(%moisture+ %volatile matter+%ash)

PROCEDUCE:-

1. Dry silica dish in an oven and weight.

2. Spread out about 1 gm of 20 mesh coal sample on the dish.

3. Weigh the dish again to find the exact mass of the sample.

4. Heat the dish without any cover in the oven at about 105-110°c for hour.

5. Take out the dish from the oven & cover it with the lid & coal.

6. Weigh the dish to find the loss in weight of coal due to presence of moisture.

Volatile matter Determination:-

1. Heat a clean crucible on its lid at 95°c for 7 minutes in muffle furnace.
2. Allow the crucible and lid to coal on a metal plate for a minute and in desiccations for 10. Minutes.
3. Weigh the Crucible and lid together.
4. Put near 1 gm sample and weigh again to know the exact mass.
5. Insert the Crucible with the lid to know the weight loss due to expulsion of volatile matter.

Ash Determination:-

1. follow the steps 1 to 3 of moisture determination.
2. Insert the open dish in the furnace at 750°c for an hour.
3. Remove the dish allow it coal for 10 minutes on the slab and 15 minutes in the desiccators.
4. Weight the dish to find the mass left which is the Ash content on the coal.

Fixed Carbon Determination:-

1. The differences between the mass of coal taken and sum of volatile Ash and moisture content give the fixed carbon content.

TABULATION:-

Sl no	Temp (c)	Time	Crucible wt (gm) A	Crucible+ coal wt(gm) B	Crucible residue(wt) C	Wt loss (B-C)
1						
2						
3						
4						
5						

CALCULATION:-

1. **Moisture:-**

Before heating at 105°c for 1hr

wt of coal + crucible= _____

After heating wt of coal +crucible=_____

Wt loss =_____

% moisture=wtloss /initial wt of coal×100=_____

2.Volatile matter:-

Before heating at 950°C for 7 minutes.

wt of coal + Crucible. = _____.

After heating wt of coal + crucible: =_____.

Wt Loss =_____.

Wt of Moisture free coal =_____

% volatile matter= wt loss/wt of moisture freecoal×100

3.ASH:-

crucible wt =_____

Before heading at 750°C

Wt of coal +crucible = _____.

Afterheating

Crucible + Residue of coal =_____

After heating out of residue Coal =_____.

% of Ash = wt of residue coal / initial wt of coal ×100

% of Fixed Carbon = 100 - (% of moisture + of volatilematter+% of Ash) = _____

CONCLUSION:-

From the above experiment we found out the percentage of moisture in coal is___%, percentage of volatile matter in coal is ____%, percentage of ash content is coal is____%,percentage of fixed content in coal is ___%.

Experiment-2

AIM OF THE EXPERIMENT

To determine the Cu% in brass by electrochemical analysis method.

APPARATUS REQUIRED:-

1. Beaker
2. Measuring cylinder
3. Conical flask
4. Digital balance
5. Electro – chemical analysis apparatus.

CHEMICAL REQUIRED

1. Concentrated nitric acid (HNO_3)
2. Concentrated sulphuric acid (H_2SO_4)
3. Potassium ferrocyanide ($C_6FeK_4N_6$)
4. Ammonia (NH_3)
5. Acetic acid (CH_3COOH)
6. Distilled water (H_2O)

SOLUTION REQUIRED

1. ***Acid solution or acid mixture:***
   30 ml concentrated nitric acid & 30ml of concentrated sulphuric acid is mixed with 40 ml distilled water to make 100 ml of acid mixture.
2. ***Potassium ferrocyanidesolution:***
   5gm of potassium ferrocyanide is dissolved in 100ml of distilled water.

PROCEDURE

- 0.5 gm of drilling of alloy was taken & dissolved in 25ml of acid mixture in a 150 ml beaker. Boil of excess below fumes & dilute to 100ml distilled water.
- The solution is electrolyzed using platinum gangue electrode Anode & cathode. A Potential of 2.5 volt was applied untilthe copper was deposited. Absence of Cu was confirm in the electrolyte.
- A drop of it was taken in a plate & a drop each it 50% ammonia of 50 % acid added to it. If copper present reddishbrown color would appear.
- The electrode was removed from the electrolyte & switching off the current. The cathode was taken out & washed with water & alcohol. Then the cathode was dried in an oven of about 105° till become dry the mixture.

- The increasing amountof weight of the cathode gave the amount of copper deposited by multiplying 100 give the percentage of Cu present in alloy.

(fig: electrochemical analysis apparatus)

CALCULATION

Initial weight of cathode (C_1)= _____

After Cu deposition weight of cathode (C_2) = _____

Increase in weight (wight of Cu deposit on cathode) = (C2-C1) = _____

% cu in 0.5 g brass= weight of Cu deposit on cathode/initial weight of brass ×100

$$= \frac{C2 - C1}{0.5} \times 100 = \_\_\_\_\_$$

CONCLUSION:

From the above experiment the percentage of copper in given sample brass is found to be _____.

Experiment -3

AIM OF THE EXPERIMENT:-

To determine the flash point and fire point of supplied liquid fuel.

APPARATUS REQUIRED:-

1- Pensky- marten
2- Supplied oil
3- Cotton
4- Match box

THEORY:

FLASH POINT:-

It is the minimum temperature at which an oil/petrofuel gives out sufficient vapor to form an inflammable mixture with air and catches-fire momentarily i.e. flashes, when flame is applied.

FIRE POINT:-

It is the lowest temperature at which vapors given off by oil ignites and continues to burn for at least 5 seconds. In most cases, fire point is 5-40°C higher than flash point and is determined in the same apparatus as for flash point determination.

It gives can idea of fire hazards during the storage and use of oil.

Pensky-Marten closed cup apparatus:

An oil cup of 5 cmdiameter and 5.5 cmdeep in which oil is filled up to the level marked inside. Four openings of standard sizes are provided in the lid of the cup. Through one of these passes a thermometer and the second opening is used for introducing test flame. Through third opening passes the stirrer carrying two brass blades. Fourth opening is meant for admission of air.

-*Shutter* is a lever mechanism, provided at the top of the cup. By moving the shutter, opening in the lid opens and flame (carried by a flame exposure device) is dipped in to this opening and brings the flame over the oil surface.

- *Flame exposure* device is a small flame and this is connected to the shutter by a lever mechanism.

- *Air bath*. Oil cup is supported by its flange over an air bath, which is heated by a gas burner.

-*Pilot burner*. As the test flame is introduced in the opening, it gets extinguished, but when the test flame is returned to its original position it is automatically lighted by the pilot burner.

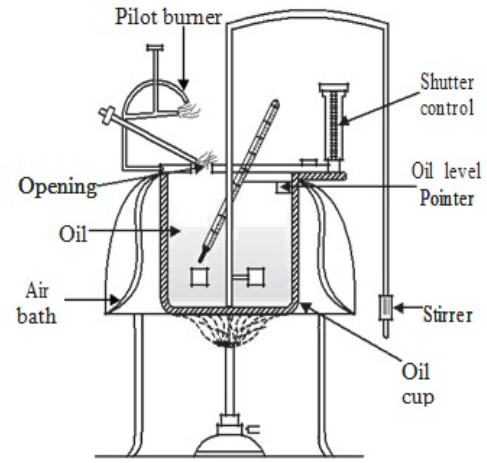

Fig. Pensky Marten's flash point apparatus

PROCEDURE:-

- The cup and its accessories are cleaned properly & dried before the test.
- Now Oil is filled up to the mark in the oil cup and it is heated by heating the air bath by a burner.
- Stirrer is worked between tests at a rate of about 1 to 2 revolutions per second.
- Heat is applied so as to raise the oil temperature by about 5°C per minute. At every 1°C rise of temperature, flame is introduced for a moment by working the shutter.
- The temperature at which a distinct flash appears inside the cup is recorded as the flash point.
- The heating is continued thereafter and the test flame is applied as before. When the oil ignites and continues to burn for at least 5 seconds, the temperature reading is recorded as the fire point of the oil.

OBSERVATION:

We observed the flash point of the liquid fuel is _____.

We observed the fire point of the liquid fuel is _____.

CONCLUSION:

From the above experiment we found out the flash point and the fire point of the supplied fuel i.e kerosene is _____ & _____ respectively**.**

Experiment-4

AIM OF THE EXPERIMENT:-

To prepare the iron ore sample and determine the iron content of that sample by potassium dichromate method.

APPARATUS REQUIRED:-

1) Burette
2) Pipette
3) Conical Flask
4) Measuring cylinder
5) Basin
6) Burette stand
7) Digital balance

CHEMICAL REQUIRED:-

- Potassium dichromate($K_2Cr_2O_7$)
- Stannous chloride (SnCl2)
- Hydrochloric acid (HCl)
- Mercury chloride (HgCl2)
- Diphenylamine sulphate (internal indicator)
- Potassium ferrocyanide($K_2[Fe_3(CN)_6]$ (external indicator)

SOLUTION REQUIRED:-

1.POTASSIUM DICHROMATE :-

-Dissolved 4.39 gm of potassium dichromate in water and make it volume of 1liter of N/10 $K_2Cr_2O_7$.

2. STANNOUSCHLORIDE:-

25gm stannous chloride is dissolved in 100ml of HCL and then make it up to ½ liter.

MERCURY CHLORIDE:-

A standard solution of Mercury chloride dissolved in water at room temperature.

It is an internal indicator which is used to drop.

THEORY:

PRINCEPLE:-

The principle involved in this titration is that only ferrous iron gets oxidized to Ferric ions and the ferric iron in the solution remains as it is.

The amount of ferrous irons present in the given solution containing ferrous and Ferricsalt is determined by titrating the solution and then the total iron (present now in the ferrous state only) is titrated with potassiumdichromate.

(1) $Cr_2O_7^{2+} + 6fe^{2+} + 14H \rightarrow 2Cr^{3+} + 6fe^{3+} + 7H2O$

(2) $2Fe^{3+} + Sn^{2+} \rightarrow 2Fe^{2+} + Sn^{4+}$

In acid solution of potassium dichromate may be represented .

$Cr_2O_7^{2+} + 14H^{+} + 6e^{-} \rightarrow 2Cr^{3+} + 7H2O$

Ferric salts are not oxidized either by dichromate permanganate. They can be determined is solution only after reduction of F_3^{3+} to F_3^{2+}ions.

The production can be carriedout by reducing agent such as $SnCl_2$ ferric salt e.gFeCl3 is reduced by the action of SnCl2 in presence of. HCL

$Fe_2(SO_4)_3 + 6HCL \rightarrow 2FeCl_3 + 3H_2SO_4$

$FeCl_3 + SnCl_2 \rightarrow 2FeCl_2 + SnCl_4$

The excess$SnCl_2$ must be remove completely as $K_2Cr_2O_7$oxidize it. This is done by adding mercuric chloride which reacts with$SnCl_2$ follow:-

$SnCl_2 + 2HgCl_2 \rightarrow SnCl_4 + HgCl_2$

(Excess added). (Silky white ppt.)

PROCEDURE:-

At Hematite, ore 0.5gm is taken by Digital balance. Then it taken in 250 ml of conical flask and is added with 10 ml of concentrated HCL, warming it upto dryness point and again 10ml of HCL is added to cold by tap water.

Then stannous Chloride solution is added drop by drop to the above iron ore solution till the red colordisappear into white.Then 10ml mercury chloride solution is added.

Then a solution of 200ml is prepared by adding water. After this we have to proceed for titration.

In a burette potassium dichromateis taken in a conical Flask 10ml of solution is taken in a glass from the above 20ml of solution.

Then the Potassium dichromate is added dropby dropand shake the solution time by time till the colure to charge to violet by the addition of barium diphenylaminesulphateto the solution.

The process is repeated up to five observation.

TABULATION:-

No of jobs	Initial Reading	Final Reading	Difference	Remark
01				
02				
03				
04				
05				

Average:-=_____=_____

CALCULATION:-

10ml of hematite solution is neutralized by _____of $K_2Cr_2O_7$ solution.

10ml of hematite solution is neutralized by _____ of $K_2Cr_2O_7$ solution

200ml of hematite neutralized by_____×__=

10ml of N/10$K_2Cr_2O_7$ solution contained= _____gm of iron

_____ml of N/10$K_2Cr_2O_7$solution contained

=_____×_____

=_____gm of iron

100ml of N/10 $K_2Cr_2O_7$ solution curtained

=_____/_____×100 =_____

CONCLUSION:-

The percentage of iron containing by supplied hematite ore was found to be _____.

Experiment -5

AIM OF THE EXPERIMENT :-

To determine the percentage of calcium and magnesium in dolomite ore.

APPARATUS REQUIRED:-

1. Conical flask
2. Heater
3. Burette Pipette -
4. Platinum crucible

CHEMICAL REQUIRED :

1. Hydrochloric Acid(1.1).
2. Ammonium oxalate (4%).
3. Ammonia Solution (1:0)
4. H_2SO_4 solution (1:10)
5. $KMnO_4$ solution

PROCEDURE:

- Dissolve 1gm of limestone in 10ml of 1:1 HCl followed by addition of few drops of NH_3 evaporate the solution to dryness and resolved in 1:1 HCl.
- Filters paper is dry and weight for Calculation of silica % for evaluation of lime content.
- The following method is adopted filtrate form the 1st part of the experiment is 250 ml with addition of dilute HCL.
- Heat the entire solution on boiling and add 4%, ammonia oxalate solution in exceeds slowly into it followed by 10 ml of ammonia solution until the whole solution in little alkaline calcium chloride by the HCL treatment. The end point of titration Indicate in the colorless solution.

CALCULATION:

Weight of the filter paper is _____.

After to filtering weight of the filter papers is _____.

Weight of Mg is _____.

Weight percentage of Mg is _____.

Initial reading of the titration is _____.

Difference = _____ - _____ = _____

1m of N/20 $KMnO_4$= _____ gm of Calcium

% of Ca = _____

% of Mg = _____

CONCLUSION:

From the experiment we found out the % of Ca and Mg in dolomite ore is ____ & _____ respectively.